Manure Sampling for Nutrient Analysis: Variability and Sampling Efficacy

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ABSTRACT

Reliable estimation of nutrient concentrations is required to manage animal manure for protecting waters while sustaining crop production. This study was conducted to investigate sample variability and reliable nutrient analysis for several manure types and handling systems. Serial samples were collected from dairy, swine, and broiler poultry operations while manure was being loaded onto hauler tanks or spreaders for field application. Samples were analyzed for total solids (TS), total nitrogen (N), ammoniacal nitrogen (NH_4–N), total phosphorus (P), and potassium (K). The least number of samples needed for reliable testing of total N and P, defined as ±10% of the experimental means with 99% probability, was obtained for each farm using a computer-intensive random resampling technique. Sample variability within farms, expressed as the coefficient of variation (CV), was mostly 6 to 8% for farms that used agitation of manure storages but several times higher (20–30%) on farms where no agitation was applied during the sampling period. Results from the random resampling procedure indicated that for farms that used agitation, three to five samples were adequate for a representative composite for reliable testing of total N and P; whereas for farms without agitation, at least 40 samples would be required. Data also suggest that using book values for manure nutrient estimations could be problematic because the discrepancies between book standards and measured farm data varied widely from a small amount to several fold.

THE essence of managing manure nutrients for reduced negative environmental impact while sustaining crop production is to control the amount of manure nutrients applied so that they meet but do not exceed crop requirements. To approach this goal, accurate knowledge of manure nutrient concentrations is a prerequisite. Published book values are available locally, often by cooperative extension agencies, or regionally and nationally, such as those by the MidWest Plan Service (1993) and USDA Natural Resource Conservation Service (1992). These book values provide average nutrient concentrations based on numerous manure test results. However, site-specific conditions on individual farms cause manure nutrient concentrations to be highly variable. Several researchers have reported substantial discrepancies between book values and farm sampling and testing data (e.g., Lindley et al., 1988; Rieck-Hinz et al., 1996; Peters, 2000). Clearly, on-farm manure sampling and nutrient testing is highly desirable.

Accurate and reliable nutrient testing depends on collecting samples representative of the manure systems. Animal manure is heterogeneous and nutrient contents of manure samples vary; thus, characterizing manure nutrient variations is a necessity for devising satisfactory sampling techniques. A number of studies have demonstrated temporal variability of manure nutrient contents on farms. From seven North Carolina dairies, Safley et al. (1985) collected monthly samples of as-excreted (i.e., fresh) and as-scraped manure (prior to entering storage) for 30 mo plus random samples of as-loaded manure (from storage onto hauler tankers for field application). With the nutrient data (TS, total N, P, K) pooled across farms and across the sampling period, the CVs ranged from 7 to 37% for as-excreted, 16 to 52% for as-scraped, and 29 to 43% for as-loaded samples (Safley et al., 1985). Rieck-Hinz et al. (1996) reported manure nutrient variability (for TS, total N, NH_4–N, P, K) for individual farms with CVs mostly in the range of 20 to 40%; the samples were obtained every 6 wk for 2 yr from dairy feedlot or dairy barn cleaner systems, or four times a year for 2 yr from bedded pack systems. Similarly, Westerman et al. (1990), based on monthly samples for a 4-yr period from storage lagoons of swine, poultry, or beef manure, found within-farm temporal variability (TS, NH_3–N, total N, P and K) with CVs ranging from 6 to 48% but mostly in the range of 10 to 20%.

The considerable temporal variability of manure nutrient contents indicates the need to sample manure near the time of field application so that nutrient applications to crops may be estimated with greater accuracy (Westerman et al., 1990). A few studies have documented manure nutrient variability with samples collected at the time of storage unloading for field applications. Muck et al. (1984), studying N conservation during manure handling and storage on two New York dairies, observed very low variability of TS, fixed solids (FS), total N, and NH_3–N with CVs of a few percent for the farm where thorough agitation was applied during storage emptying. For the other farm where storage unloading was by outlet pipe with little mixing, lack of uniformity was illustrated graphically (Muck et al., 1984). O'Dell et al. (1995) presented graphical data demonstrating up to threefold differences in N and P concentrations from manure samples collected over the course of applying swine manure from an anaerobic lagoon to experimental plots. In a Dutch study comparing several sampling methods for slurry or solid manure during storage unloading, Derikx et al. (1997) reported CVs of log-transformed data of dry matter (DM), total N, P, and K in the range of 8 to 47%, with most data points between 20 and 30%.

Data from most of the studies indicate considerable variability of manure nutrients and suggest the need for determining nutrient contents based on multiple samples. To minimize analysis cost, it is generally recommended to collect multiple subsamples and combine them into a single composite sample for laboratory test-

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Published in J. Environ. Qual. 30:1432–1437 (2001).

Abbreviations: TS, total solids.

ing. However, a key issue remaining to be resolved is the number of subsamples needed to make a representative composite that yields satisfactory results. Our literature search yielded only one data source addressing the relevant issue (Iversen et al., 1997; K.V. Iversen, personal communication, 2000). The researchers, with samples collected from selected manure stockpiles in Colorado and means and CVs calculated based on 10 subsamples per stockpile, used the equation $n_{est} = t^2\,CV^2\,p^2$ to estimate the number of subsamples (n_{est}) needed for obtaining nutrient results within the 95% confidence interval with a 10% probable error (p). Calculated n_{est} varied substantially depending on the target nutrients (N, P, or K) and the type of manure stockpile. For example, to obtain total N, P, and K within the 95% confidence intervals with a 10% probable error, the estimated n_{est} would be 1, 5, and 119, respectively, for dairy compost; 55, 31, and 27 for chicken manure; or 17, 20, and 32 for a beef manure stockpile.

Still, more research data are needed to address the concerns of whether or not it is practical to collect manure samples for reliable nutrient testing, and if the answer is yes, how many subsamples are needed to make a representative composite that would provide nutrient results with statistically defined accuracy and reliability. In the present study, serial samples (n = 10 to 35) were collected from several commercial farms during manure storage unloading for field application. The nutrient concentrations were determined for individual samples and the data were then analyzed for the purposes of (i) characterizing nutrient variability for the individual manure systems and (ii) estimating the least number of subsamples needed for the given systems for accurate and reliable results with statistically defined goals.

MATERIALS AND METHODS

To facilitate discussion, manure is operationally defined in the present study as the material that was loaded from the storage to hauler tanks or a spreader for field application at the time the samples were collected, regardless of animal species (dairy, swine, or poultry), storage type and physical attributes (such as liquid or solid), or inclusion and/or exclusion of other materials (bedding, feed refusal, barnyard runoff, etc.). This definition avoids potential confusion associated with the use of ambiguous phrases such as slurry, effluent, semi-solid, solid waste, animal waste, farm yard manure, etc.

Farm Information and Sample Collection

Manure samples were collected from seven commercial farms in southeastern Pennsylvania including dairy, swine, and broiler poultry operations (Table 1). The five dairies all had Holstein cows, fed with farm-grown forages and high-moisture corn plus purchased concentrates. Ration balancing services were provided by feed supply company personnel. Each farm had either aboveground tank or in-ground pit manure storage, which was emptied two or three times a year by the owner or by a commercial hauler, with the manure applied to owned or rented cropland. Manure applications were made mostly to corn fields prior to planting and in some cases to forage fields after the first and/or second hay harvest. Main differences between farms in terms of manure handling and other relevant information are summarized in Table 1.

Sample collection on the dairy operations took place in spring 1999 during the course of manure storage unloading for field application. Sampling followed a systematic approach with serial samples taken at regular intervals from the beginning to the end of the storage unloading period. Sampling intervals varied from every load on one farm to every third load on another depending on estimated manure volume, anticipated load numbers, and time span of the hauling and spreading processes. Unforeseeable changes in weather and other farming activities occurred, resulting in different sample numbers and time spans for sample collections on the individual farms. In some cases, the manure storage was only partially emptied (Table 1). For Farm D-Pit2, each sample consisted of several hand-grab samples from the skid loader while loading manure onto a box spreader. On the other four dairies where manure was pumped to hauler tanks, samples were obtained at the end of hauler tank loading from either the tip of the loading hose or the opening on the top of the hauler tank. Each raw sample was placed in a plastic bucket from which approximately 500 g was taken after mixing.

Table 1. Manure storage, handling, source, and sampling information for seven commercial operations in southeastern Pennsylvania. B-Mix and B-Floor samples were from two broiler houses on the same operation.

Farm ID	Manure storage and handling	Other information	Number of samples
D-Tank1	Dairy, aboveground tank storage; 36-h agitation prior to and during storage unloading; completely emptied by pumping.	70 lactating cows; the manure tank also contained milk house waste and outside lot runoff water; spring sampling from alternate loads over 3 d.	35
D-Tank2	Dairy, aboveground tank; 24 h agitation prior to and during storage unloading; partially emptied by pumping.	400 lactating cows; the manure tank also contained milk house and parlor waste; spring sampling from every third load over 2 d.	35
D-Tank3	Dairy, aboveground tank; agitated during storage unloading; nearly emptied by pumping.	70 lactating cows; the tank also contained milk house waste; spring sampling from every load over 1 d.	20
D-Pit1	Dairy, in-ground pit storage; 5-h agitation prior to and during storage unloading; partially emptied by pumping.	45 lactating cows; the pit also contained manure from a heifer barn and milk house waste; spring sampling from alternate loads over 3 d.	10
D-Pit2	Dairy, in-ground pit storage; no agitation; nearly emptied.	150 lactating cows; the pit also contained manure from a heifer barn, milk house waste plus loafing yard runoff; spring sampling from every third load over 2 wk.	11
Swine	Swine lagoon (in-ground); no agitation; not emptied.	1200 breeding sows and 720 gilts; fall sampling from every load over 2 d.	20
B-Mix	Broiler house litter mounded for hauling; completely emptied.	Two or three cycles of 25 000 birds per cycle per house; sawdust bedding added between cycles; spring sampling in 1 d.	31
B-Floor	Broiler house litter, floor samples.	Same as B-Mix.	32

The swine samples were collected from a breeding operation of approximately 1200 sows and 720 gilts, housed in individual crates or small grouped pens on slatted floors. Manure and wastewater entered a concrete pit under the pens. The pit was flushed periodically and manure flowed to an outside storage lagoon with plastic liner. While much of the solids settled and accumulated at the bottom of the lagoon, the liquid portion was reduced (never emptied) three times a year by pumping to hauler tanks and spread onto cropland. To avoid damaging the lagoon liner, no agitation had ever been used during the eight years the lagoon had been in place, including during the present study (fall 1999). Sampling followed the same protocol as for the dairy farms.

The two sets of poultry samples were collected from two broiler houses on a single farm. The broiler houses, each measuring 152 by 15 m, were cleaned twice a year after two or three production cycles. Each cycle consisted of eight weeks of bird rearing (approximately 25 000 birds per house), from day-old chicks to market weight, followed by a week preparing for the next cycle. Sawdust as bedding was added between each cycle. Sampling took place during the broiler house cleaning in spring 1999. The B-Mix set was from one house, where the litter had been pushed to the end of the barn by a skid loader. Random samples were collected during a 4-h period while the litter was being removed. For each sample, several hand-grab samples were taken near the loading dock and placed in a bucket from which about 250 g was obtained after thorough mixing. The B-Floor sample set was from the floor of a second house before litter removal. Sixteen samples were obtained from each side of the barn at equally spaced points along the barn and approximately 3.6 m from the exterior wall. Litter samples from about a 65-cm^2 area were removed from the floor and placed in a bucket, mixed, and approximately 250 g of subsample was taken.

The serial samples for each farm were labeled in the order they were collected. All samples were immediately placed in a portable cooler and transferred to our laboratory by the end of the day. Samples were stored frozen (−25°C) until further processing.

Sample Analysis

To prepare sample material for laboratory analysis, manures were thawed, and a representative subsample was taken and homogenized in a blender to pass a 3-mm screen. All samples were analyzed for total solids (TS), total N, P, K, and ammoniacal N. Total solids were determined by drying at 60°C overnight in a gravity air oven. For total N, P, and K determinations, a subsample equivalent to 0.25 g dry matter was digested in 5 mL H_2SO_4 and 15 to 30 mL H_2O_2 at 440°C (adaptation of Method 3050B; USEPA, 1995). Total N was determined using Nessler's reagent (Method 4500-NH_3 C; American Public Health Association, 1992). Total P and K were determined using inductively coupled plasma–atomic emission spectroscopy (ICP–AES) (Method 6010B; USEPA, 1995). Ammonium N concentration was determined using Nessler's reagent after mixing the manure subsample with deionized water.

Statistical Methods

Least square means, standard deviations, and CVs of TS, total N, P, K, and NH_4–N were calculated for each farm or sample set using SAS (SAS Institute, 1996). A computer-intensive random resampling technique was used to determine the number of samples required for reliable estimates of total N and P for each farm. This is a nonparametric technique in that no assumptions are required concerning the underlying population distribution (detailed discussions on the statistical and theoretical specifics may be found in Efron [1982] and Diaconis and Efron [1983]). According to Starr et al. (1992) who employed this technique for the determination of soil sample numbers required for reliable testing of NO_3–N, the number of resamples in the subsets drawn from the original data set is not constrained by the number of observations in the original data set. In their study, Starr et al. (1992) also compared this technique with the conventional approach, the equation described by Petersen and Calvin (1986) and modified by Iversen et al. (personal communication, 2000). Their soils data show that sample numbers calculated using the equation were overestimated in the experiments with intensive sampling in a small area, and underestimated in the experiments with larger-scale area. The researchers concluded that where sample variance and frequency distribution can be only approximately estimated, the random resampling technique should produce more accurate estimates than the Petersen and Calvin method (Starr et al., 1992).

With the computer-intensive random resampling technique, a subset of resamples was randomly drawn from the original data set for each farm with each resample returned to the original set before the next drawing; the means of total N and P of each subset were obtained. This procedure was repeated 10 000 times for each subset and the percentage of the means falling within ±5, 10, and 15% of the experimental means (mean of the original data set) were recorded. For each farm, the size of the subset started with three resamples; the number of resamples in the subset were increased until the means of total N and P of the last subset fell within ±10% of the experimental means 99% of the time.

RESULTS AND DISCUSSION

Sample Variability

Summary statistics for TS, total N, NH_4–N, P, and K computed for each farm are presented in Table 2. Sample variability within farms, as indicated by the CV values, was generally 6 to 8% for the farms where agitation was applied (D-Tank1, D-Tank2, D-Tank3, D-Pit1, and B-Mix). However, where no agitation was used (D-Pit2, Swine, and B-Floor), sample variability was several times greater, with the relevant CVs mostly in the range of 20 to 30%. Evidently, agitation during storage unloading, even the mechanical push-up in the broiler case (B-Mix), greatly reduced the heterogeneity of manure. This suggests that much of the nutrient variability within manure storage is in a macro instead of a micro scale and can be reduced substantially through agitation or mixing.

Variability in manure nutrient concentrations obtained in the present study through intensive and serial sampling is comparable with literature reports. With agitation, CV values were mostly <10% (present study and Muck et al., 1984). Without agitation, CV values in the present study (20–30%) were similar to the findings by Derikx et al. (1997). Also, sample variability in the present study as well as Derikx's (Derikx et al., 1997), in which samples were collected during manure storage unloading, appeared to be in a similar range compared with studies where samples were from different times within farms (CVs of 20–40% were reported by Rieck-

Hinz et al., 1996; 10–30% reported by Westerman et al., 1990). Interestingly, a comparable range of nutrient variability was also found for samples from different farms with similar storage and manure type: CVs of 20 to 30% reported by Lindley et al. (1988); 20 to 40% reported by Safley et al., 1985; and 10 to 30% obtained for the five dairies in the present study.

The magnitude of sample variability within farms in the present study, even for those with no agitation (20–30%), is in a range comparable with soil nutrient variability in agronomic studies such as fertilizer rate trials. Peterson and Krueger (1980) recorded CVs of 10 to 20% for soil-available P and approximately 10% for available K during an 8-yr fertilizer rate trial. Starr et al. (1992) reported CV values for soil nitrate concentrations ranging from 28 to 162% depending on sample size and sampling area. While agronomic research typically uses replications and composites of multiple samples within replications as the way to minimize and quantify variability so that treatment differences may be detected, for manure sampling and nutrient testing, much of the sample variability would be eliminated with reasonable agitation, as suggested by the data in the present study. The underlying message is that manure sampling for nutrient analysis should be advocated similarly to soil nutrient testing to encourage environmentally responsible management of manure.

Nevertheless, nutrient concentrations on any given farm display a rather wide range, even for those with agitation and relatively low variability (last column in Table 2). For example, for D-Tank1, there was a 20 to 30% difference between the measured minimum and maximum of total N, NH_4–N, P, and K (Table 2). A close examination of the data for the serial samples revealed little consistency in terms of the sequence of samples and the relevant analyses: the minimums were not necessarily associated with the samples taken at the beginning of the storage unloading period, nor the maximums with those at the end of period. Clearly, multiple samples are needed in order to obtain reliable nutrient results even for manure systems with relatively low variability.

Table 2. Summary statistics for total solids (TS, %), total N (g kg^{-1}), ammoniacal nitrogen (NH_4–N, g kg^{-1}), and total phosphorus and potassium, (P, K, g kg^{-1}) for manure samples from commercial operations.

Farm ID	Measurement	Mean	Standard deviation	CV	Minimum–maximum
D-Tank1	TS	6.70	0.41	6.16	5.82–7.64
(n = 35; agitated)	total N	3.10	0.20	6.00	2.80–3.60
	NH_4–N	1.50	0.10	6.16	1.40–2.00
	P	0.48	0.04	7.28	0.39–0.57
	K	1.58	0.08	4.34	1.41–1.74
D-Tank2	TS	8.31	0.96	14.06	5.74–10.59
(n = 34; agitated)	total N	3.60	0.20	4.86	3.20–4.00
	NH_4–N	1.90	0.15	7.92	1.50–2.20
	P	0.65	0.06	9.26	0.52–0.78
	K	1.49	0.11	6.72	1.33–1.83
D-Tank3	TS	10.28	0.63	6.11	9.14–11.32
(n = 20; agitated)	total N	5.00	0.20	3.27	4.70–5.20
	NH_4–N	2.40	0.20	6.20	2.20–2.80
	P	0.92	0.04	6.86	0.78–1.05
	K	2.99	0.08	3.63	2.82–3.24
D-Pit1	TS	5.59	0.83	14.90	4.36–6.93
(n = 10; agitated)	total N	3.40	0.20	6.90	3.10–3.70
	NH_4–N	2.00	0.10	6.50	1.80–2.20
	P	0.57	0.09	15.4	0.48–0.74
	K	2.16	0.08	4.8	1.99–2.32
D-Pit2	TS	13.85	3.05	22.0	7.27–18.15
(n = 11; no agitation)	total N	5.20	1.60	30.11	2.50–8.60
	NH_4–N	1.90	0.40	19.55	1.30–2.50
	P	0.92	0.22	24.14	0.48–1.31
	K	2.99	0.42	14.19	2.32–3.57
Swine	TS	0.61	0.07	16.69	0.30–0.74
(n = 21; no agitation)	total N	1.70	0.23	13.63	1.10–2.10
	NH_4–N	1.50	0.24	16.02	0.89–1.90
	P	0.14	0.03	23.92	0.08–0.21
	K	1.00	0.23	22.52	0.64–1.38
B-Mix	TS	66.50	2.42	3.5	61.9–70.9
(n = 35)	total N	30.20	2.00	6.2	26.00–35.00
	NH_4–N	5.75	0.75	12.3	4.00–8.00
	P	11.73	1.05	8.5	9.26–13.52
	K	16.10	1.41	8.7	14.11–18.26
B-Floor	TS	68.83	9.30	13.6	50.4–86.0
(n = 32)	total N	26.90	9.00	33.3	10.00–43.00
	NH_4–N	6.00	2.30	37.9	3.00–11.00
	P	8.98	2.96	32.9	2.62–13.52
	K	16.35	2.99	18.0	10.79–21.58

Sampling Efficacy

Results using the computer-intensive random resampling technique are presented in Table 3, expressed as the frequency that the means of total N and P for each resampling subset fall within ±5, 10, 15% of the experimental means. Considering 99% probability (i.e., 99%

Table 3. Percentage of time that random resample means fall within ±5, 10, and 15% of the original means for total N and P for manure samples from commercial operations.

Farm ID	Resample no.	Total N 5	Total N 10	Total N 15	P 5	P 10	P 15
		%					
D-Tank1	3	86.4	99.7	100.0	76.4	98.8	100.0
(agitated)	5	94.8	100.0	100.0	88.5	99.8	100.0
D-Tank2	3	85.6	98.9	99.9	65.6	94.9	99.8
(agitated)	5	93.3	99.8	100.0	78.7	99.0	100.0
D-Tank3	3	99.6	100.0	100.0	79.3	98.9	100.0
(agitated)	5	100.0	100.0	100.0	90.3	100.0	100.0
D-Pit1	3	81.9	99.4	100.0	43.5	77.0	92.2
(agitated)	5	91.5	100.0	100.0	56.7	88.3	98.3
	10	98.7	100.0	100.0	71.9	97.1	99.9
	15	99.7	100.0	100.0	81.0	99.3	100.0
D-Pit2	3	26.1	53.8	68.8	36.8	56.8	75.0
(no agitation)	5	32.2	57.7	75.9	38.2	67.6	85.8
	10	41.4	73.5	90.5	50.9	83.2	96.3
	20	56.4	88.4	98.0	67.1	95.0	99.6
	30	66.3	94.3	99.6	76.4	98.3	100.0
	40	72.9	97.1	99.9	82.8	99.3	100.0
	50	78.6	98.6	100.0	87.3	99.8	100.0
	60	82.4	99.3	100.0	90.5	99.9	100.0
Swine	3	51.5	80.8	93.6	26.9	54.1	74.9
	5	60.2	89.9	98.8	36.7	66.7	85.2
	10	76.3	98.0	100.0	50.4	82.6	95.6
	20	89.3	99.9	100.0	65.7	94.6	99.6
	30	95.2	100.0	100.0	75.5	98.1	100.0
	40	98.2	100.0	100.0	82.2	99.3	100.0
B-mix	3	52.0	83.2	95.7	66.3	95.8	99.7
	5	91.9	99.9	100.0	80.7	98.9	100.0
	10	98.6	100.0	100.0	93.3	100.0	100.0
B-floor	3	20.2	37.6	53.8	18.8	39.6	55.4
	5	26.2	50.0	68.8	26.8	50.5	68.9
	10	36.5	65.9	85.0	36.9	66.5	85.8
	20	49.9	82.2	96.2	50.8	83.3	96.1
	30	59.4	90.3	98.8	60.1	90.8	99.0
	40	66.4	94.7	99.7	66.6	94.9	99.7
	50	72.0	97.0	99.9	72.8	97.2	99.9
	60	76.5	98.3	100.0	76.9	98.5	100.0
	70	79.9	98.8	100.0	80.6	98.9	100.0
	75	81.2	99.2	100.0	82.1	99.2	100.0

Table 4. Means of total solids (TS), total N, and total phosphorus (P) in manure, as compared with selected book values.

Manure type	Data source	TS	Total N	P
		%	g/kg	
Dairy	MWPS (liquid pit)†	8	3.875	1.875
	PA (solid)‡	13	5.0	2.0
	PA (liquid)	<5	3.5	1.6
	Present study			
	D-Tank1	6.7	3.1	1.1
	(means ± 10%)		(2.8–3.4)	(1.0–1.2)
	D-Tank2	8.3	3.6	1.5
	(means ± 10%)		(3.2–4.0)	(1.35–1.65)
	D-Tank3	10.3	5.0	2.1
	(means ± 10%)		(4.5–5.5)	(1.89–2.31)
	D-Pit1	5.6	3.4	1.3
	(means ± 10%)		(3.1–3.7)	(1.17–1.43)
Swine	MWPS (lagoon)	1	0.625	0.375
	NRCS (anaerobic lagoon)§	0.25	0.364	0.0788§
	PA (anaerobic lagoon, supernatant)	0.25	0.364	0.0788§
	Present study	0.61	1.65	0.31
	(means ± 10%)		(1.48–1.82)	(0.28–0.34)
	(means ± 15%)		(1.28–1.90)	(0.26–0.36)
Broiler	MWPS (with bedding)	75	23.5	24.0
	NRCS (with bedding)	76	19.4	9.7
	PA (heavy broiler, no bedding)	25	33.0	31.5
	Present study			
	B-mix	66.5	30.2	26.9
	(means ± 10%)		(27.2–33.2)	(24.2–29.6)
	(means ± 15%)		(25.7–34.7)	(22.9–30.9)

† MidWest Plan Service (1993), Tables 10-6, 10-7.
‡ Penn State College of Agricultural Science (1998), Table 2-15.
§ USDA Natural Resources Conservation Service (1992), Table 4-13 and Table 4-15 in Chapter 4.

of the time) as the criterion for reliability, data in Table 3 show that three to five samples would make a composite with means of total N and P falling within ±10% of the experimental means for the farms that applied agitation, including the B-Mix samples. This is comparable, with statistically defined reliability and accuracy, with the multiregional manure sampling guidelines reported in Peters (2000).

For those manure storages that received no agitation, the number of samples needed for reliable nutrient testing increased dramatically: for D-Pit2, 40 to 60 samples would be required to achieve an accuracy of ±10% of the experimental means; for the B-Floor sample set, 75 samples are required to meet the ±10% criterion (Table 3). In the Swine case, 20 samples are required for total N and 40 samples for P in order to achieve the ±10% accuracy (Table 3). Apparently, the number of samples needed for accurate (within ±10% of experimental means) and reliable (99% probability) nutrient testing is nearly impractical as a routine practice on farms if no agitation is applied. The multiregional manure sampling guidelines reported by Peters (2000) do not recommend sampling for bedded packs or unagitated liquid manure storage facilities.

Inaccurate nutrient analysis resulting from inadequate sampling can have serious agronomic production or environmental consequences. For example, if sampling and analysis data deviate by 15% from the actual means, the resultant application would vary by ±54 kg N ha^{-1} of manure assuming a corn crop with a net N requirement of 125 kg ha^{-1} and fertilizer equivalence of 0.35 for the total N in manure.

Measured Data versus Book Values

The experimental means of nutrients in the present study can be viewed as reliably representing the relevant manure systems. Hence, such data provide an opportunity for meaningful comparisons against standard book values. Data from D-Pit2 and B-Floor were excluded from this comparison exercise because they might be inadequate as representative samples, as suggested by the random resampling results.

Discrepancies between the experimental means of total N and P and local (Penn State College of Agricultural Science, 1998), regional (MidWest Plan Service, 1993), or national (USDA Natural Resources Conservation Service, 1992) standards vary a great deal (Table 4). For the swine and broiler manures, none of the book values fall within ±10% and most are not within ±15% of the experimental means of total N or P. Discrepancies are in both directions (i.e., under- and overestimations). Also, there appears to be as much difference among the three book standards as between the experimental means and the book values (Table 4). It should be noted that the book values derived from MidWest Plan Service (1993) and USDA Natural Resources Conservation Service (1992) in Table 4 are for broiler manure with bedding (which is the case in this study) while the PA standards (Penn State College of Agricultural Science, 1998) are for broiler manure without bedding.

For the dairy manure, the accuracy of nutrient estimates using book standards depends on which set of book values are chosen. For example, for D-Tank2, the PA (liquid) standards would be satisfactory (within ±10% of the experimental means) while the PA (solid) standards would result in greater than +15% deviation from the experimental means (Table 4). For D-Tank3, the reverse is true: the PA (solid) standards would be accurate while the PA (liquid) values would result in more than −15% deviation from the experimental means (Table 4). There has been a lack of consistency, or rather a great deal of confusion, associated with the physical categorization of manure. For example, the PA standards designate dairy manure of <5% TS as *liquid*, 13% TS as *solid*; the MidWest Plan Service (1993) specifies TS of 8% as *liquid pit*; while according to USDA Natural Resources Conservation Service's (1992) classification, TS of 0 to 2%, 3.5 to 7.5%, 10 to 14%, and >16% corresponds to liquid, slurry, semi-solid, and solid for dairy manure, respectively. For field personnel or farmers using the PA standards, the challenge is to choose the "right" set of book values when sample TS falls between the two sets of standards (>5 but <13%).

Book values as averages of many samples from different sources can provide useful references in whole-farm nutrient planning, such as estimating annual generation of manure nutrients and the corresponding cropland requirement. However, to guide field application practices, manure sampling and reliable testing are required.

CONCLUSION

Manure nutrient variability within manure handling systems appears to be largely macro scale, which can be substantially reduced through reasonable efforts of agitation or mixing during storage unloading. Accurate and reliable nutrient analysis is achievable with three to five samples for the agitated systems included in the present study. Sampling from nonagitated manure systems may offer limited chance for satisfactory nutrient measurements unless extensive sampling, in the magnitude of 40 or more samples, is conducted. Standard book values may provide useful information for whole-farm nutrient planning, but could be problematic for guiding field manure application practices.

ACKNOWLEDGMENTS

The authors are grateful to all participating farmers for their cooperation during sample collection. This project was partially supported by the Pennsylvania Department of Agriculture, Agriculture Research Program.

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